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(58) Field of search G2J
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(54) Chromatographic flow cell

(57) A flow cell 10 for use in a UV absorbance detector comprises an irradiation cavity or aperture 16 of cylindrical or disc shape. The cavity is defined by (a) transparent irradiation windows 18, 20 spaced apart in the direction of irradiation by a distance which is small compared with the width of the face of the windows, and (b) an annular peripheral wall, and inlet and outlet ducts 34, 36 which connect with the cavity through ports in the peripheral wall. The ports are disposed in the circumferential direction of the wall, the ports being positioned and the ducts being orientated so as to provide when a fluid from a chromatographic column is flowing therethrough turbulent flow in the cavity and good cavity scavenging and window scrubbing action.

ERRATUM

SPECIFICATION NO 2089062A

Front page, Heading (72) Inventors *for* Warran *read* Warren

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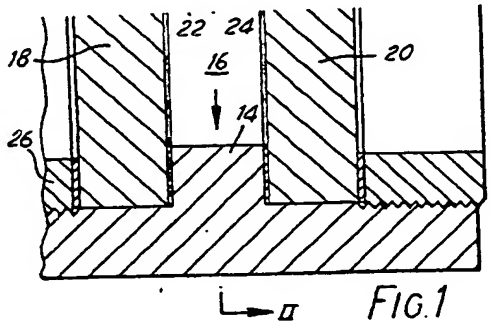

FIG.1

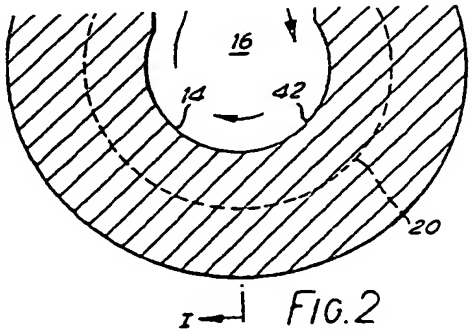

FIG.2

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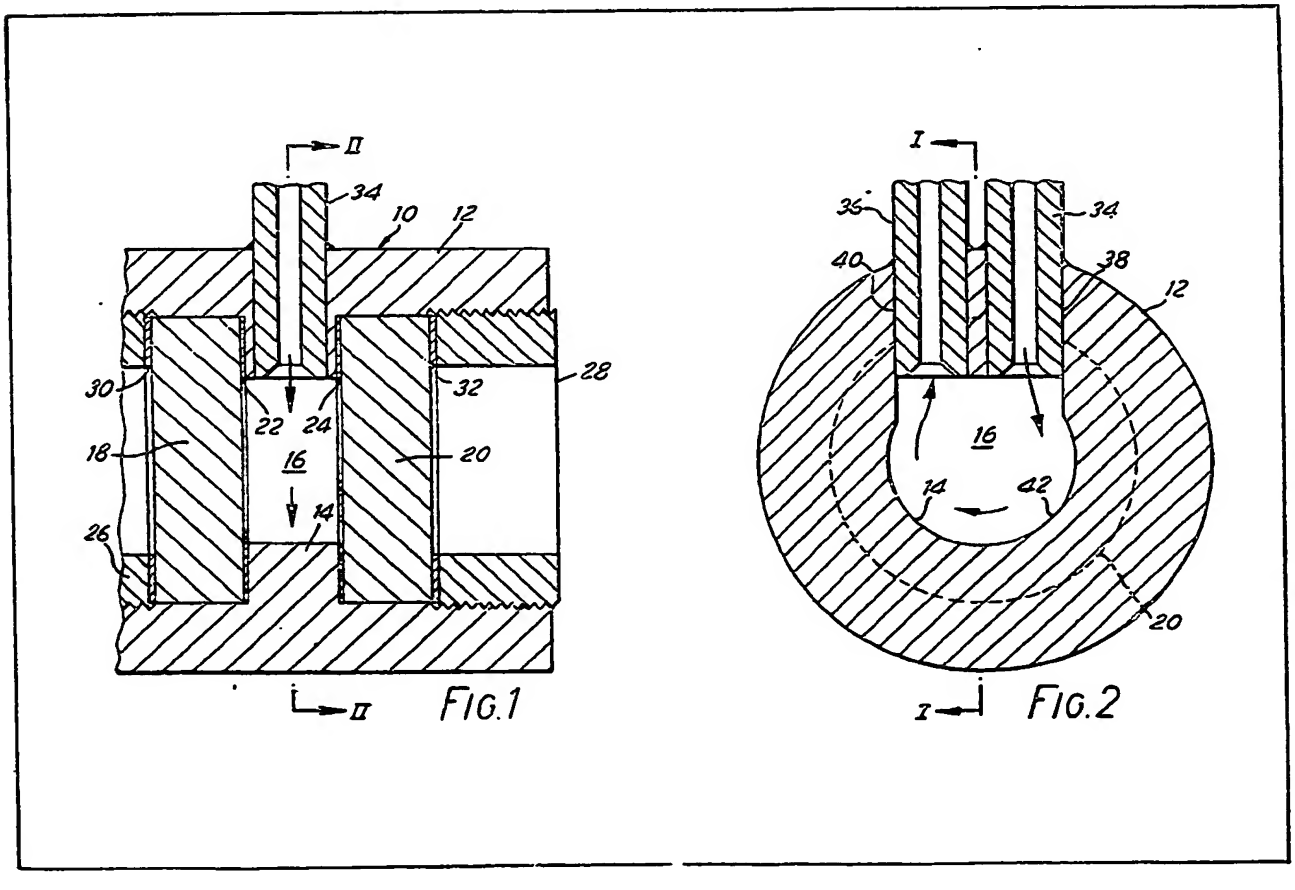

FIG.1 FIG.2

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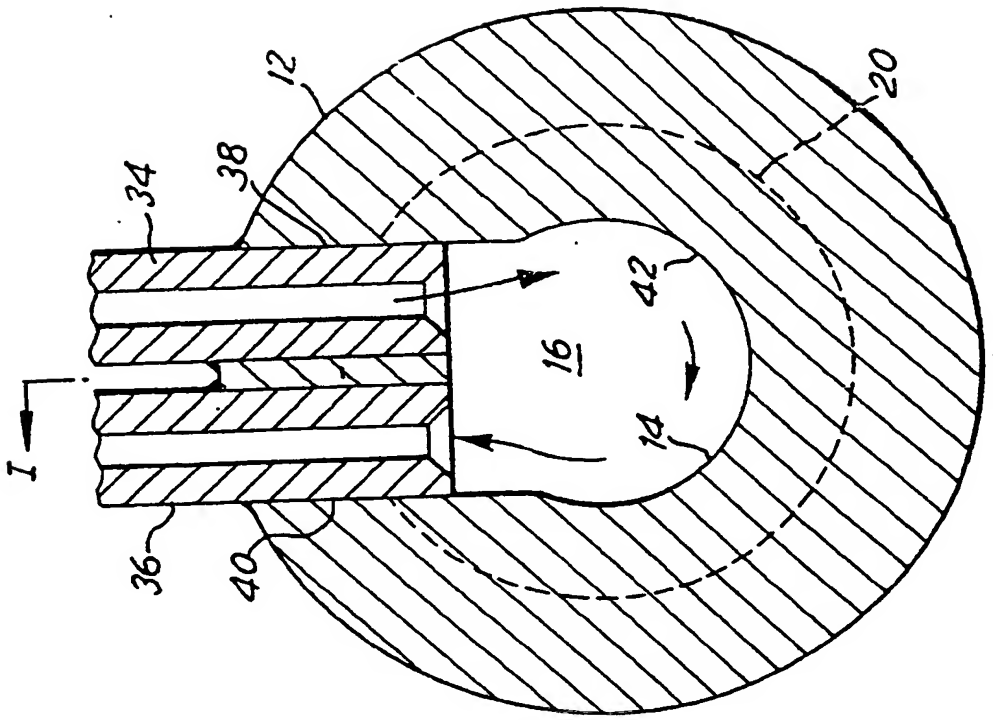

FIG.2

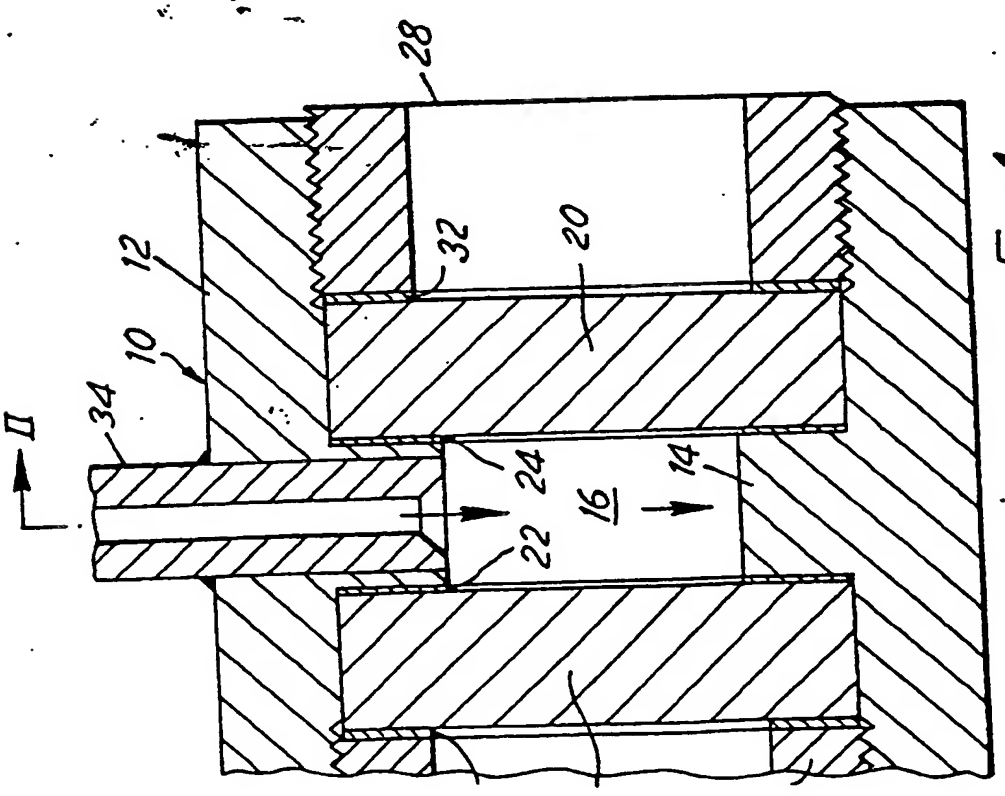

FIG.1

SPECIFICATION

Chromatographic apparatus

This invention relates to chromatographic apparatus. Such apparatus may be used for analysing a small sample of a mixture of substances (analytical work) to determine its respective constituents, or for separating out and retrieving a particular constituent or constituents in a larger sample of a mixture of substances (preparative work).

In carrying out preparative work it is necessary as a preliminary activity to carry out a certain amount of analytical work in order to optimise the parameters of the chromatographic column, and to determine from the chromatograms so obtained the time period or periods in which the particular desired constituent or constituents is or are to be collected from the apparatus when doing the preparative work.

Hitherto, both the analytical work and the preparative work have been carried out in succession using the same detector for the two, substantially differently sized chromatographic columns, and to accommodate the greatly increased column flow rate (for a common mobile phase velocity) when the detector has been used with the preparative column, a bypass has been fitted to the detector so as to bypass a high proportion of the output of the column. Furthermore, with the fairly high concentration in the preparative sample of the desired constituent(s) to be collected, the detector could provide only very low outputs, since the detector had been designed to give good results with the type of sample normally being examined in analytical work, that is to say samples with low concentration of the substances being looked for.

A detector typically includes a transparent flow cell through which the output of the column can flow either to waste, or to a collector vessel, the flow cell being arranged so that the contents thereof may be irradiated by a beam of UV or other light (or other EM radiation), and the effect of the contents on a said beam being detected by a detecting device arranged to receive radiation emerging from said flow cell.

In a typical UV absorbance detector, a beam of UV light passes through said cell and its contents, and the emergent beam activates a UV sensitive device so as to cause it to provide a signal dependent on the amount of UV light absorbed by the contents of the flow cell.

A commonly used flow cell for such a UV detector has a long slender irradiation duct aligned with the beam of light and having inlet and outlet ducts connected at the respective ends thereof. The three ducts are connected in a Z-configuration so that the irradiation duct is thoroughly and continuously scavenged by the fresh liquid flow entering from the chromatographic column, and so that the irradiation windows at the respective transverse ends of the irradiation duct are thoroughly scrubbed. Moreover, the irradiation duct has a wall which is slightly divergent in the direction of the outlet so as to encourage the liquid flow in that duct to be laminar. The length of the irradiation duct is made quite small (typically 8 mm) so as to provide a good length of sample in which the UV light can be absorbed, thus providing high sensitivity; and the diameter of that duct is made quite small (typically 1 mm) so that a highly concentrated beam of UV light can be used to irradiate the sample currently in the flow cell.

The present invention seeks to provide a substantially different form of flow cell for use particularly in a UV absorbance detector, which form of cell can be used with both the analytical and the preparative chromatographic columns without the need for bypass circuits such as have been referred to earlier in the specification, and which can provide satisfactory chromatograms (with adequate and satisfactory sensitivities) for both the analytical work and the associated and subsequent preparative work.

According to the present invention a flow cell for use in a UV absorbance detector suitable for both analytical and preparative work has an irradiation cavity or aperture of cylindrical or disc shape, the cavity being defined by (a) circular transparent irradiation windows spaced apart in the direction of irradiation by a distance which is small compared with the diameter of the windows, and (b) an annular peripheral wall, and inlet and outlet ducts which connect with said cavity through ports in said peripheral wall, which ports are disposed adjacent one another in the circumferential direction of said wall, the ports being positioned and the ducts being oriented so as to provide when a liquid from a chromatographic column is flowing therethrough turbulent flow in the cavity and good cavity scavenging and window scrubbing action.

Other features and advantages of the present invention will appear from the description that follows hereafter.

One flow cell according to the present invention for a UV absorbance detector will now be described by way of example and with reference to the accompanying drawings in which:—

Figure 1 shows a longitudinal vertical section through the flow cell, taken on the section I-I of Figure 2; and

Figure 2 shows a transverse vertical section through the flow cell, taken on the section II-II of Figure 1.

Referring now to the drawings, the flow cell 10 comprises a stainless steel tubular body 12 in which an integral central web 14 defines a cylindrical central irradiation aperture 16. The central web is enclosed on either side by thick, plane irradiation windows 18, 20 of quartz or silica, and annular PTFE seals 22, 24 are located between those windows and the central web to ensure liquid-tightness under pressure. The windows are held in position by stainless steel annular, screw-threaded retaining rings 26, 28 which are screwed into the internally screw-threaded ends of the body 12 and which bear on said

windows via externally placed PTFE seals 30, 32.

Stainless steel inlet and outlet tubes 34, 36 have their respective free ends brazed into two parallel holes 38, 40 which penetrate at central locations the wall of the body 12 and also said central web 14. The tube ends extend to the cylindrical wall 42 defining said irradiation aperture or cavity 16.

The disc-shaped irradiation aperture 16 has an irradiation length of 2 mm and a diameter of 4 mm. The inlet and outlet tubes have bores of 0.5 mm nominal. The construction shown and described is made to withstand ordinary working pressures of 500 pounds per square inch, and test and other momentary pressures of up to 2000 pounds per square inch.

As will be seen from Figure 2, the direction of liquid flow in the irradiation aperture is generally parallel to the cylindrical boundary wall 42 defining that aperture. In tests, with preparative-work flows of up to 50 mls/min the liquid flow in the irradiation cavity has been fully turbulent (there being at no position in the cavity a stagnant body of liquid), and such as to give good self-scavenging action and good scrubbing of the windows. Hence, in operation each later separated substance has not been contaminated by a remnant of an earlier separated substance. Thus, good resolution has been obtained, with various injected input samples.

The flow cell is intended to be used with a UV light beam that irradiates the whole transverse cross-section area of the irradiation cavity.

Despite the greatly reduced irradiation length of sample in the flow cell, the sensitivity obtained when performing preparatory analytical work, with the output of an analytical column, has been quite good, being as much as one third of that obtainable with the aforesaid flow cell with a Z-shaped liquid flow path. That sensitivity has been wholly adequate and satisfactory for performing the necessary analytical work as a preliminary to the subsequent preparative work.

A UV absorbance detector equipped with a test flow cell as described above is capable of working at wavelengths of 200 to 650 nm (or even up to 800 nm) and giving absorptions in the range 0.05 to 5.0 absorption units full scale deflection, with mobile phase flow rates of up to 50 ml/min. (or even up to 100 ml/min.) and operating pressures up to 500 or more pounds per square inch.

The positioning and orientation of the inlet and outlet ducts is capable of variation within certain limits, providing the necessary turbulence and freedom from stagnant volumes in the cavity are obtained, though preferably the ducts should be placed as near as possible to the respective peripheral sides of the cavity.

CLAIMS (filed 3 Dec 1981)

1. A flow cell for use in a UV absorbance detector comprising an irradiation cavity or aperture of cylindrical or disc shape, the cavity being defined by (a) transparent irradiation windows spaced apart in the direction of irradiation by a distance which is small compared with the width of the face of the windows, and (b) an annular peripheral wall, and inlet and outlet ducts which connect with said cavity through ports in said peripheral wall, which ports are disposed in the circumferential direction of said wall, the ports being positioned and the ducts being orientated so as to provide when a fluid from a chromatographic column is flowing therethrough turbulent flow in the cavity and good cavity scavenging and window scrubbing action.

2. A flow cell according to Claim 1 in which the ducts are placed as near as possible to the respective peripheral sides of the cavity.

3. A UV absorbance detector when equipped with a flow cell according to Claim 1 or 2.

4. A flow cell substantially as hereinbefore described with reference to the accompanying drawings.

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